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Indian Standard

PLASTICS — DETERMINATION OF THE ULTIMATE ANAEROBIC BIODEGRADATION AND DISINTEGRATION UNDER HIGH-SOLIDS ANAEROBIC-DIGESTION CONDITIONS — METHOD BY ANALYSIS OF RELEASED BIOGAS

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NEW DELHI 110002

March 2009 **Price Group 5**

Plastics Sectional Committee, PCD 12

NATIONAL FOREWORD

This Indian Standard which is identical with ISO 15985 : 2004 'Plastics — Determination of the ultimate anaerobic biodegradation and disintegration under high-solids anaerobic-digestion conditions — Method by analysis of released biogas' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Plastics Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker in the International Standard while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

The technical committee responsible for the preparation of this standard has reviewed the provision of the following International Standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 8245 : 1999	Water quality — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PLASTICS — DETERMINATION OF THE ULTIMATE ANAEROBIC BIODEGRADATION AND DISINTEGRATION UNDER HIGH-SOLIDS ANAEROBIC-DIGESTION CONDITIONS — METHOD BY ANALYSIS OF RELEASED BIOGAS

1 Scope

This International Standard specifies a method for the evaluation of the ultimate anaerobic biodegradability of plastics based on organic compounds under high-solids anaerobic-digestion conditions by measurement of evolved biogas and the degree of disintegration at the end of the test. This method is designed to simulate typical anaerobic digestion conditions for the organic fraction of mixed municipal solid waste. The test material is exposed in a laboratory test to a methanogenic inoculum derived from anaerobic digesters operating only on pretreated household waste. The anaerobic decomposition takes place under high-solids (more than 20 % total solids) and static non-mixed conditions. The test method is designed to yield the percentage of carbon in the test material and its rate of conversion to evolved carbon dioxide and methane (biogas).

The conditions described in this International Standard may not always correspond to the optimum conditions for the maximum degree of biodegradation to occur.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8245, *Water quality — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

3 Terms and definitions

3.1
ultimate anaerobic biodegradation
breakdown of an organic compound by microorganisms in the absence of oxygen to carbon dioxide, methane, water and mineral salts of any other elements present (mineralization) plus new biomass

3.2
disintegration
physical breakdown of a material into very small fragments

3.3
total dry solids
amount of solids obtained by taking a known volume of test material or inoculum and drying at about 105 °C to constant mass

3.4

volatile solids

amount of solids obtained by subtracting the residue of a known volume of test material or inoculum after incineration at about 550 °C from the total dry solids content of the same sample

NOTE The volatile solids content is an indication of the amount of organic matter present.

3.5

lag phase

time, measured in days, from the start of a test until adaptation and/or selection of the degrading microorganisms is achieved and the degree of biodegradation of a chemical compound or organic matter has increased to about 10 % of the maximum level of biodegradation

3.6

maximum level of biodegradation

degree of biodegradation, measured in per cent, of a chemical compound or organic matter in a test, above which no further biodegradation takes place during the test

3.7

biodegradation phase

time, measured in days, from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached

3.8

plateau phase

time, measured in days, from the end of the biodegradation phase until the end of the test

4 Principle

The test method is designed to be an optimized simulation of an intensive anaerobic digestion process and determines the ultimate biodegradability and degree of disintegration of a test material under high-solids anaerobic digestion conditions. The methanogenic inoculum is derived from anaerobic digesters operating on pretreated household waste, preferably only the organic fraction.

The test material is mixed with the inoculum and introduced into a static digestion vessel where it is intensively digested under optimum temperature and moisture conditions for a test period of 15 days or longer until a plateau in net biodegradation has been reached.

During the anaerobic biodegradation of the test material, methane, carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass) are the ultimate biodegradation products. The biogas (methane and carbon dioxide) evolved is continuously monitored or measured at regular intervals in test and blank vessels to determine the cumulative biogas production. The percentage biodegradation is given by the ratio of the amount of biogas evolved from the test material to the maximum theoretical amount of biogas that can be produced from the test material. The maximum theoretical amount of biogas produced is calculated from the measured total organic carbon (TOC). This percentage biodegradation does not include the amount of carbon converted to new cell biomass which is not metabolized in turn to biogas during the course of the test.

Additionally, the degree of disintegration of the test material is determined at the end of the test and the loss in mass of the test material may also be determined.

5 Test environment

Incubation shall be in the dark or in diffused light in an enclosure that is maintained at a constant temperature of 52 °C ± 2 °C.

6 Reagents

Use only analytical-grade reagents.

Use TLC (thin-layer chromatography) grade cellulose with a particle size of less than 20 µm as the positive-control reference material.

7 Apparatus

Ensure that all glassware is thoroughly cleaned and, in particular, free from organic or toxic matter.

Required is usual laboratory equipment, plus the following:

7.1 Digestion vessels: conical or other suitable glass flasks connected up so that no loss of gas occurs.

A minimum volume of 750 ml is recommended in view of the requirements of 8.2 and 8.3.

Weigh each empty digestion vessel if the loss in mass of the test material is to be determined.

7.2 Gas volume measurement system, comprising an inverted graduated cylinder or plastic column in water or another suitable device for measuring gas volume. The water in contact with the gas shall be at a pH of less than 2 during the whole period of the test to avoid CO_2 loss through dissolution in the water. The gas volume measuring device, as well as the gas tubing, shall be of sufficient quality to prevent gas migration and diffusion between the system and the surrounding air.

7.3 Apparatus for gas analysis (optional), comprising a gas chromatograph, or other apparatus, equipped with a suitable detector and column(s) for measuring the methane and carbon dioxide concentration in the evolved gases.

7.4 Analytical apparatus (optional), for determining volatile fatty acids by aqueous-injection chromatography, as well as total Kjeldahl nitrogen, ammonia nitrogen, dry solids (at 105 °C) and volatile solids (at 550 °C).

8 Procedure

8.1 Preparation of the inoculum

The inoculum shall be obtained from a properly operating anaerobic digester using pretreated household waste as the sole feedstock. The pretreated household waste should preferably come from an existing waste treatment facility that is treating municipal solid waste where, through sorting, shredding, sieving or other means, a fairly homogeneous organic fraction is produced with a particle size of less than 60 mm.

The digester should have been operating for a period of at least 4 months using this organic fraction, with a retention time of a maximum of 30 days under thermophilic conditions (52 °C ± 2 °C). Gas evolution yields should be at least 15 ml of biogas at standard temperature and pressure per gram of dry solids in the digester and per day on average for at least 30 days.

The inoculum should preferably be derived from a digester operating under dry (> 20 % total solids) conditions. It can, however, also be derived from a wet fermentation process, the digested sludge being dewatered by centrifugation, by using a press or by drying at a maximum temperature of 55 °C to a total solids content of at least 20 %.

The prepared inoculum should undergo a short post-fermentation of approximately 7 days at the same operating temperature as that of the facility from which it was derived. This means that the inoculum is not fed but allowed to postferment anaerobically by itself. This is to ensure that large, easily biodegradable particles are degraded during this period and also to reduce the background level of degradation of the inoculum itself.

The most important biochemical characteristics of the inoculum shall be as follows:

- pH: between 7,5 and 8,5;
- volatile fatty acids (VFA): below 1 g/kg of wet mass;
- ionized-ammonia nitrogen (NH_4^+-N): between 0,5 g/kg and 2 g/kg of wet mass.

8.2 Preparation of test material and reference material

Determine the total organic carbon (TOC) in the test material and the reference material using e.g. ISO 8245 and report it preferably as grams of TOC per gram of total dry solids. Alternatively, if the materials do not contain inorganic carbon, it is possible to determine the carbon content by elemental analysis. The test material shall have sufficient organic carbon to yield methane and carbon dioxide in amounts suitable for the determination. Normally, a minimum of 20 g of total dry solids containing 8 g of TOC is required per vessel.

If the loss in mass is to be determined, determine the total dry solids and volatile solids of the test material.

NOTE The loss in mass of the test material and reference material during the test can be determined, optionally, as additional information. In the example given in Annex B, the volatile-solids content of the test material is determined at the beginning of the test and compared with that at the end of the test.

Use test material in the form of granules, powder, film or simple shapes (e.g. dumb-bells). The maximum surface area of any individual piece of test material shall be about 2 cm × 2 cm. If any pieces in the original test material are large, reduce them in size.

8.3 Start-up of the test

Set up at least the following number of digestion vessels (7.1):

a) three vessels for the test material;

b) three vessels for the reference material;

c) three vessels for blank controls.

Remove enough inoculum (approximately 10 kg) from the post-fermentation vessel and mix carefully and consistently by hand in order to obtain a homogeneous medium.

To every 1 000 g of wet mass (at least 20 % dry solids) of inoculum, add an amount of test material or reference material containing 15 g to 20 g of volatile solids for each test vessel. Use a minimum amount of 500 g of wet mass of inoculum per test vessel. Mix the test mixture manually in a small container for a period of 2 min to 3 min.

Treat the three blanks, containing inoculum only, in similar manner — by manually mixing the inoculum in a small container for a period of 2 min to 3 min with the same intensity as was done for the other vessels containing test material or reference material.

Place the mixtures in the digestion vessels and gently spread and compact the material evenly in the vessels to a uniform density. Put the vessels in a water bath or incubator and connect them to the gas-measurement or gas-collection device. The length of time between mixing the inoculum with the test or reference material and connection to the gas-measurement system should not be longer than 2 h. Record the temperature and atmospheric pressure prior to turning on the heating system of the incubator or water bath.

If loss in mass measurements are to be carried out, accurately weigh and record the mass of inoculum and test material placed in each individual digestion vessel. If the test material is in the form of shapes, they may be retrieved at the end of the digestion period to evaluate the degree of disintegration.

8.4 Incubation period

Incubate the digestion vessels in the dark or in diffused light for a nominal period of 15 days at a constant temperature of 52 °C ± 2 °C, which is representative of full-scale anaerobic digestion. The incubation time can be extended until a constant plateau phase is reached, if significant biodegradation of the test material is still observable.

Measure the volume of biogas evolved at different time intervals to obtain a plot of evolved biogas volume as a function of time. More frequent measurements in the early stages of the experiment may be required.

8.5 Termination of the test

At the end of the digestion period, allow the digestor vessels to cool to room temperature and determine the total volume of biogas evolved during the test. Record the room temperature and atmospheric pressure.

If the loss in mass of the test material is to be determined, weigh the digestor vessels with their test mixtures. Take samples of the test mixtures from all vessels. Determine the total dry solids and the volatile solids (see Annex B).

Record any visual observations with regard to the appearance of the test material to determine its degree of disintegration.

Optionally, the methane and carbon dioxide (biogas) concentrations can be measured by using analytical apparatus suitable for the detection and quantification of these gases, such as a gas chromatograph with an appropriate detector.

It is recommended that further investigations be carried out with any test material remaining such as measuring relevant physical properties, chemical analysis and taking photographs.

9 Calculation and expression of results

9.1 Calculation of gaseous carbon

First, the amount of gaseous carbon C_g evolved from each digestion vessel is calculated. The volumes of methane and carbon dioxide evolved are converted to volumes at standard conditions of temperature (273 K) and pressure (1 013,25 hPa) (= STP) using the ideal-gas equation:

$$\frac{pV}{T} = \text{constant} \tag{1}$$

where

p is the pressure in hPa;

V is the volume in litres;

T is the temperature in K.

These volumes are also corrected for water vapour pressure and atmospheric pressure variations during the test.

The corrected volume of biogas evolved is converted to the corresponding amount of gaseous carbon evolved using the standard equation: 22,4 ml of biogas at STP = 12 mg of C_g.

9.2 Calculation of the percentage biodegradation

Calculate the mean (of the three replicates) net amount, in grams, of gaseous carbon evolved by anaerobic biodegradation of the test material by subtracting the mean amount, in grams, of gaseous carbon evolved from the controls (three replicates) containing only inoculum.

Calculate the percentage biodegradation using the following equation:

$$\%\ \text{biodegradation} = \frac{m_{\mathrm{C,g}}(\text{test}) - m_{\mathrm{C,g}}(\text{blank})}{m_{\mathrm{C,i}}} \times 100 \tag{2}$$

where

$m_{\mathrm{C,g}}$ is the amount of gaseous carbon evolved, in grams;

$m_{\mathrm{C,i}}$ is the amount of carbon initially in the test material, in grams.

Calculate the standard error σ_{M} of the percentage biodegradation as follows:

$$\sigma_{\mathrm{M}} = \sqrt{\left[\frac{\sigma^2(\text{test})}{n_1} + \frac{\sigma^2(\text{blank})}{n_2}\right]} \times \frac{100}{m_{\mathrm{C,i}}} \tag{3}$$

where

n_1 and n_2 are the number of replicate test and blank digesters, respectively;

σ_{M} is the standard deviation of the total amount of gaseous carbon produced.

Use the same equation to calculate the percentage biodegradation of the reference material.

9.3 Calculation of loss in mass

An example of the optional calculation of loss in mass based on volatile-solids content is given in Annex B.

9.4 Expression of results

Compile tables containing the measured and calculated data on the test material, the reference material and the blank controls.

Plot the cumulative amount of biogas evolved from each digestion vessel (blank, test material and reference material) as a function of time. Plot a biodegradation curve (percentage biodegradation as a function of time) for the test material and the reference material. Use mean values if the differences between the individual values are less than 20 %. If this is not the case, plot biodegradation curves for each digestor vessel.

Read from the plateau phase of the biodegradation curve the mean degree of biodegradation and report it as the final test result.

If the test material consisted of discrete pieces, describe qualitatively the degree of disintegration of the material. Add further information such as photographs or measured values of relevant physical properties, if available.

10 Validity of results

The test is considered as valid if:

a) the percentage biodegradation of the reference material is more than 70 % after 15 days;

b) the difference between the percentage biodegradation of the reference material in the different vessels is less than 20 % of the mean value.

11 Test report

The test report shall provide all pertinent information, particularly the following:

a) a reference to this International Standard;

b) all information necessary to identify and describe the test material, such as dry- or volatile-solids content, organic-carbon content, shape and visual appearance;

c) all information necessary to identify and describe the reference material and its organic-carbon content;

d) the volume of the digestor vessels, the amounts of inoculum, test material and reference material used and the main characteristics of the equipment used to determine the volume of biogas produced;

e) information on the inoculum, such as source, age, date of collection, storage, handling, stabilization, total dry solids, volatile solids, pH of suspension, total nitrogen content and volatile fatty acids, as appropriate;

f) the results obtained for the biogas evolution and percentage biodegradation for each digestor vessel and the averages, in tabular form and graphically, as well as the final percentage biodegradation for the test material and the reference material and the activity of the inoculum;

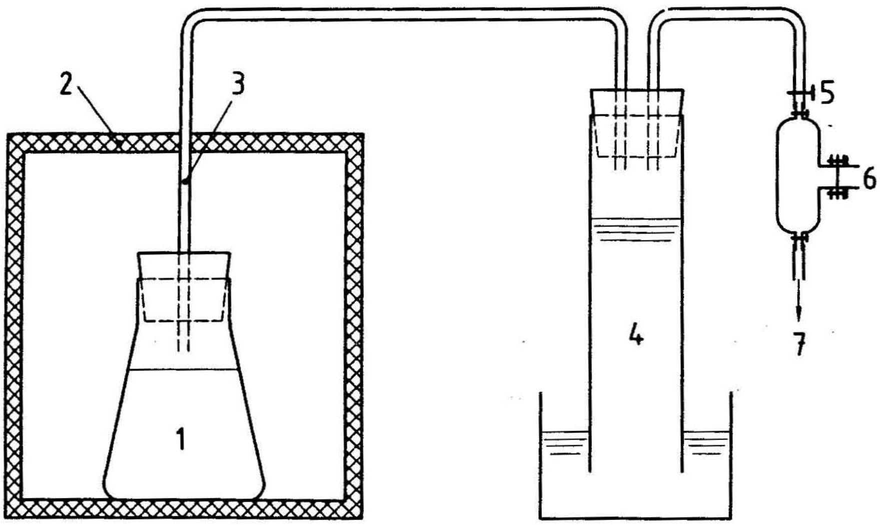
g) the results of visual observations on the test material and inoculum during and at the end of the test, such as degree of disintegration, physical measurements and/or photographs;

h) the mass of each digestor vessel at the start and the end of the test and details of any loss in mass measurements, if performed;

i) the reasons for rejection of any test results.

Annex A
(informative)

Principle of test system



Key

1 digester
2 incubator
3 gas outlet
4 gas collector
5 valve
6 gas sampling
7 gas discharge

Figure A.1 — Apparatus for high-solids anaerobic-digestion test

Annex B
(informative)

Example of loss in mass determination

Determine and calculate the percentage of mass loss of the test material based on volatile-solids content using the following procedure. Use the same procedure for the reference material.

$$\text{Mass loss from replicate } i\ (\%) = \frac{b_i}{a_i} \times 100 \qquad \text{(B.1)}$$

where

a_i is the mass of the volatile solids in the test material in replicate i at the start (g/vessel);

b_i is the loss in mass of volatile solids from the test material in replicate i (g/vessel).

Thus, the mass loss from replicate i (%) is given by

$$[(w_{t1,i} \times d_{t1,i} \times v_{t1,i}) - (w_{t2,i} \times d_{t2,i} \times v_{t2,i})] - [(w_{b1} \times d_{b1} \times v_{b1}) - (w_{b2} \times d_{b2} \times v_{b2})] \qquad \text{(B.2)}$$

where

$w_{t1,i}$ is the wet mass in test vessel i at the start (g/vessel);

$w_{t2,i}$ is the wet mass in test vessel i at the end;

w_{b1} is the average wet mass in the blank vessels at the start;

w_{b2} is the average wet mass in the blank vessels at the end;

$d_{t1,i}$ is the dry solids in test vessel i at the start (% relative to the wet mass);

$d_{t2,i}$ is the dry solids in test vessel i at the end;

d_{b1} is the average dry solids in the blank vessels at the start;

d_{b2} is the average dry solids in the blank vessels at the end;

$v_{t1,i}$ is the volatile solids in test vessel i at the start (% relative to the dry solids);

$v_{t2,i}$ is the volatile solids in test vessel i at the end;

v_{b1} is the average volatile solids in the blank vessels at the start;

v_{b2} is the average volatile solids in the blank vessels at the end.

Bibliography

[1] ISO 625:1996, *Solid mineral fuels — Determination of carbon and hydrogen — Liebig method*

[2] ISO 11734, *Water quality — Evaluation of the "ultimate" anaerobic biodegradability of organic compounds in digested sludge — Method by measurement of the biogas production*

[3] ISO 14855, *Determination of the ultimate aerobic biodegradability and disintegration of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide*

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